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MINES BRANCH INVESTIGATION REPORT IR 72 - 19

FLOTATION OF CARBON FROM GREAT CANADIAN OIL SANDS FLY ASH

A. STEMEROWICZ AND R. W. BRUCE

MINERAL PROCESSING DIVISION

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Mines Branch Investigation Report IR 72-19

FLOTATION OF CARBON FROM GREAT CANADIAN OIL SANDS FLY ASH

by

A. Stemerowicz* and R.W. Bruce**

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SUMMARY OF RESULTS

The fly ash which is a waste product from the burning of pulverized petroleum coke contains 39.8% carbon, 1.4% vanadium and 0.66% nickel. It is composed of two types of particles; amorphous carbon grains and amorphous alumino-silicate spheroids which contain varying amounts of vanadium and nickel.

Pilot plant flotation of carbon with kerosene as collector and pine oil and Dowfroth 250 as frothers gave results as follows:

	Analysis, %			Distribution, %		
	C	V	Ni	C	V	Ni
Carbon conc	84.1	0.40	0.16	99.7	14.1	10.8
Flotation tailing	0.21	2.02	1.10	0.3	85.9	89.2

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INTRODUCTION

Source of Sample

The Great Canadian Oil Sands Ltd. plant is located at Tar Island near Fort McMurray, Alberta. The fly ash is a waste product from the burning of pulverized petroleum coke in boilers which produce steam for electric power generation and for the separation process.

The coke used as fuel in the boilers is the solid carbonaceous product produced when the bitumen is subjected to the coking process which is the first stage in the refining of the bitumen to synthetic crude oil. Vanadium, nickel and residual mineral particles which are present in the bitumen are concentrated in the coke. When the coke is burned, vanadium and nickel are further concentrated in the fly ash. The present fly ash production has a potential recovery of 1,200,000 lb of V_2O_5 and 500,000 lb of nickel annually.

Shipment

On June 23, 1971 a shipment consisting of 2250 lb of fly ash in steel drums was received by Mr. G.E. Viens, Head of the Pyrometallurgy Section, Metals Reduction and Energy Centre. It was submitted by Bechtel and Company, consultants for Great Canadian Oil Sands Limited.

Nature of Investigation

The sample was submitted for the purpose of investigating smelting techniques for the recovery of vanadium and nickel. Before any of these techniques could be applied the high carbon content of the fly ash had to be reduced. This could be done by roasting the fly ash at a high temperature but energy requirements were high as were the losses of fine material in the

waste gases. In a discussion with Mr. Viens regarding this problem it was suggested that flotation might be a more economical method for reducing the carbon content of the fly ash and that the Non-Ferrous Minerals Section of the Mineral Processing Division would be willing to carry out some preliminary testing.

When bench-scale tests showed that flotation was a feasible method for removing carbon it was requested that 4 drums of fly ash (about 1100 lb) be processed in the pilot plant in order to provide sufficient low-carbon material to continue the smelting tests. A request was also made to investigate the possibility of removing silica by flotation. Because of high flux requirements in smelting any reduction in silica content would be beneficial.

Sampling and Analysis

It was stated that the fly ash was uniform in characteristics and therefore riffling of the available material was not necessary in order to obtain a representative sample for bench-scale testing. Instead, a 10-lb sample was scooped out of one of the drums. The analysis of the fly ash as given by Mr. Viens is shown in Table 1.

TABLE 1

Analysis of Fly Ash

	%
Loss on ignition	42.9
Carbon (C)	39.8
Vanadium (V)	1.4
Nickel (Ni)	0.66
Iron (Fe)	4.11
Copper (Cu)	0.0053
Sulphur (S)	2.16
Phosporus (P)	0.03
Silicon (Si)	11.6
Aluminum (Al)	7.73
Calcium (Ca)	2.09
Magnesium (Mg)	0.61

Fe_2O_3 588
SiO_2 2481
Al_2O_3 1461
CaO 292
MgO 101
Total 9328
not counting V and Ni oxides

DETAILS OF INVESTIGATION

Bench-Scale Carbon Flotation

A 1000-gram sample of fly ash was repulped with tap water in a 1000-gram D-1 Denver laboratory flotation cell and a carbon concentrate was floated off with kerosene as the collector and pine oil and Dowfroth 250 as frothers. The carbon concentrate was upgraded by cleaning and recleaning in a 1000-gram cell. Results and reagents and conditions for flotation are given in Tables 2 and 3 respectively.

TABLE 2

Results of Bench-Scale Carbon Flotation

Product	Weight %	Analysis, %*				Distribution, %			
		C	V	Ni	Fe	C	V	Ni	Fe
Carbon conc	27.7	86.5	0.31	0.13	0.32	58.8	6.5	5.4	2.4
Cleaner tail No. 2	4.2	73.6	0.65	0.29	1.07	7.6	2.1	1.8	1.2
Cleaner tail No. 1	17.1	61.4	0.85	0.41	1.96	25.9	11.0	10.7	9.1
Rougher tail	51.0	6.2	2.08	1.06	6.32	7.7	80.4	82.1	87.3
Feed (calcd)	100.0	40.7	1.32	0.66	3.69	100.0	100.0	100.0	100.0
Feed (assay)		39.8	1.4	0.66	4.11				
1st stage cl conc (calcd)	31.9	84.8	0.35	0.15	0.42	66.4	8.6	7.2	3.6
Rougher conc (calcd)	49.0	76.6	0.53	0.24	0.96	92.3	19.6	17.9	12.7

*By Mineral Sciences Division, Internal Report MS-AC-71-446.

TABLE 3

Reagents and Conditions for Bench-Scale Carbon Flotation

Operation	Time min	% Solids	pH	Reagents, lb/ton		
				Kerosene	Pine oil	Dowfroth 250
Conditioning	2	19	4.4	0.12	0.16	0.072
Rougher flotation						
Stage 1	2			0.12		0.036
" 2	1			0.12		
" 3	2			0.12		0.036
" 4	2					
No. 1 Cleaner		10				0.036
Stage 1	1					0.036
" 2	3					
No. 2 Cleaner		7				0.036
Stage 1	1					0.036
" 2	2					

Pilot-Plant Carbon Flotation

Three pilot plant runs were carried out as follows:

Run No.	Date	Operating time	Sampling period
1	Nov. 9, 1971	9.00 a.m. - 4.00 p.m.	12.00 noon - 4.00 p.m.
2	Nov. 10, "	10.30 a.m. - 4.00 p.m.	12.30 p.m. - 4.00 p.m.
3	Nov. 12, "	9.00 a.m. - 4.00 p.m.	10.30 a.m. - 4.00 p.m.

The fly ash was fed at a rate of 60 lb/hour along with 0.5 g.p.m. water to give a flotation feed density of 17% solids.

In Run No. 1, not enough of the carbon was floated from the fly ash with the result that the tailing still contained 21% C. This gave a recovery of 64% in a carbon concentrate containing 92.2% C.

For Runs No. 2 and 3 the rougher flotation capacity was increased from 1.5 cu ft to 3.6 cu ft and scavenger flotation cells were added to give an additional 1.5 cu ft of flotation capacity. Also, feed rates for kerosene, pine oil and Dowfroth 250 were increased. These changes resulted in a reduction in carbon content in the tailing to about 0.25% C. It is estimated that about 400 lb of low-carbon tailing were produced in Runs No. 2 and 3.

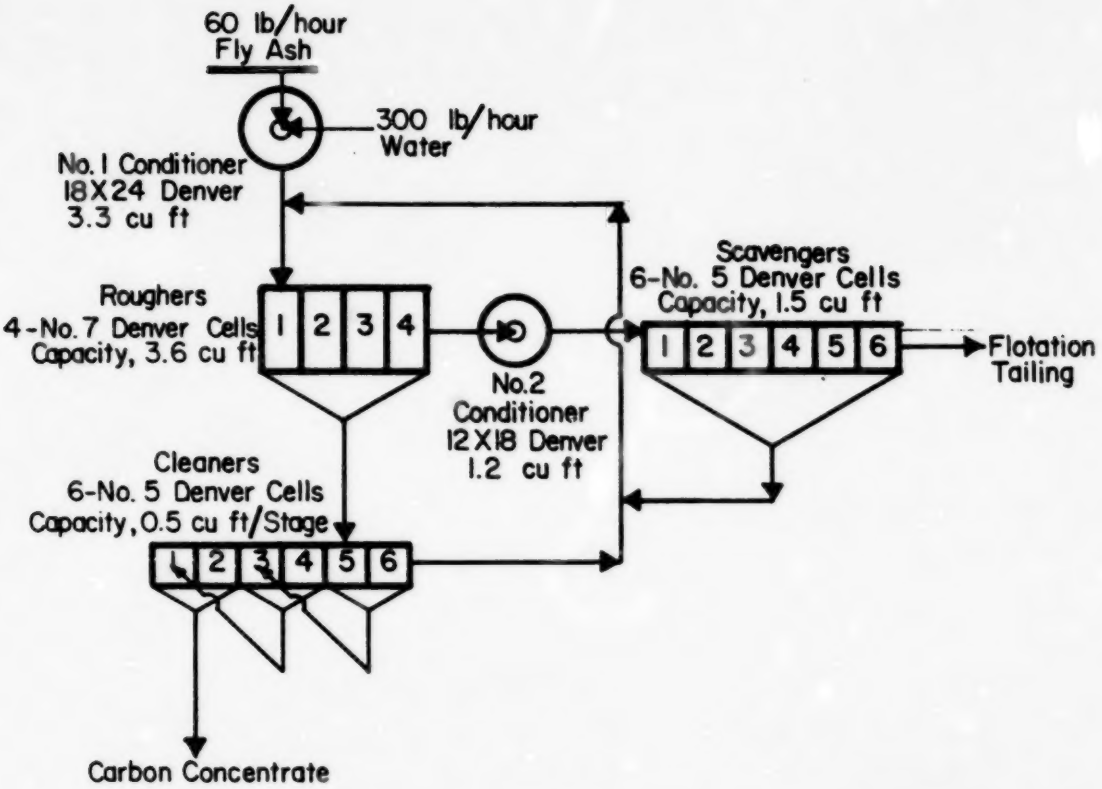
Results for Run No. 3 are given in Table 4 while the flowsheet is shown in Figure 1.

TABLE 4

Results of Carbon Flotation, Pilot Plant Run No. 3

Product	Weight* %	Analysis, %**										
		C	V	Ni	Fe	Total S	S/SO_4	Si	Ca	Mg	Al	LOI
Carbon conc	45.4	84.1	0.40	0.16	1.7	1.19	0.02	1.90	0.28	0.094	1.33	90.0
Tailing	54.6	0.21	2.02	1.10	6.9	0.05	0.02	20.1	3.03	1.01	13.7	0.52
Feed (calcd)	100.0	38.3	1.28	0.67	4.5	0.57	0.02	11.8	1.78	0.59	8.08	41.1
Feed (assay)	100.0	38.3	1.31	0.64	4.1	0.59	0.01	11.9	1.91	0.55	7.91	42.5
		Distribution, %										
Carbon conc		99.7	14.1	10.8	17.0	95.2	45.5	7.3	7.1	7.2	7.5	99.3
Tailing		0.3	85.9	89.2	83.0	4.8	54.5	92.7	92.9	92.8	92.5	0.7
Feed (calcd)		100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

*Carbon analyses of products used in calculating weight per cent.
**By Extraction Metallurgy Division.



REAGENT FEED RATES, lb/ton

Point Of Addition	Pine Oil	Kerosene	Dowfroth 250
No. 1 Cond	0.62	0.49	
No.1 Ro Cell			1.63
No.3 Ro Cell		0.31*	0.31*
No.2 Cond		0.53	
No. 1 Scav Cell			0.03
No.3 Scav Cell		0.56	
No.4 Scav Cell			0.03
Total	0.62	1.89	2.00

* Fed as 1:1 mixture of Kerosene and Dowfroth 250 emulsified in water.

Figure 1. PILOT PLANT FLOWSHEET FOR CARBON FLOTATION

Silica Flotation

Two batch scale tests were tried in which attempts were made to float a silica concentrate from the fly ash after carbon flotation. The American Cyanamid reagent, Aeroamine 3037 was used as the collector and Dextrine 4356 (Stein-Hall, Limited) was added to improve selectivity. Results of the one test for which the products were analysed is given in Table 5 followed by reagents and conditions in Table 6.

TABLE 5

Results of Silica Flotation

Product	Weight %	Analysis, %*				Distribution, %			
		C	V	Ni	Si	C	V	Ni	Si
Carbon rougher conc	52.2	72.0	0.73	0.32	4.80	93.3	26.4	27.4	20.6
Silica conc	4.3	8.51	1.95	0.86	18.90	0.9	5.8	6.1	6.7
Silica cleaner tail	3.9	15.50	1.92	1.06	16.90	1.5	5.1	6.7	5.3
Final tailing	39.6	4.34	2.28	0.92	20.70	4.3	62.7	59.8	67.4
Feed (calcd)	100.0	40.3	1.44	0.61	12.18	100.0	100.0	100.0	100.0

*By Extraction Metallurgy Division.

TABLE 6

Reagents and Conditions for Silica Flotation*

Operation	Time min	% Solids	pH	Reagents, lb/ton feed		
				Dextrine 4356	Aeroamine 3037	Dowfroth 250
Conditioning	5		9.0**	2.0		
Rougher flotation		10				
Stage 1	1				0.28	0.035
Stage 2	2					0.035
Cleaner	2	6		0.5		

*Reagents and conditions for preceding carbon flotation similar to that given in Table 3.
**Natural pH after acid salts in fly ash removed by water wash prior to carbon flotation.

Size Analysis

In order to determine metal distribution by size, minus 250-mesh samples of fly ash and low-carbon flotation tailing were run through a Warman Cyclosizer. The fly ash was also sized by passing the material through 200, 400 and 500-mesh Tyler screens. Results are given in Tables 7, 8 and 9.*

*Chemical analysis by Extraction Metallurgy Division.

TABLE 7

Metal Distribution in Fly Ash Screen Fractions

Size Fraction	Weight %	Analysis, %				Distribution, %			
		C	V	Ni	Si	C	V	Ni	Si
+200 mesh	6.8	86.0	0.22	0.09	1.6	14.3	1.0	1.0	0.9
-200, +400 mesh	24.4	83.0	0.36	0.16	3.3	49.6	6.2	6.5	6.6
-400+500 mesh	19.7	56.0	1.24	0.57	10.8	27.0	17.2	18.7	17.3
-500 mesh	49.1	7.56	2.19	0.90	18.8	9.1	75.6	73.8	75.2
Total (calcd)	100.0	40.8	1.42	0.60	12.3	100.0	100.0	100.0	100.0
Total (assay)		38.3	1.31	0.64	11.9				

TABLE 8

Metal Distribution in Fly Ash Cyclosizer Fractions

Size Fraction	Normal Size* Microns	Weight %	Analysis, %				Distribution, %			
			C	V	Ni	Si	C	V	Ni	Si
+250 mesh		13.7	82.0	0.28	0.10	1.95	29.3	2.9	2.1	2.2
No. 1 cone u'flow	+41	1.0								
No. 2 " "	+31	8.3	73.0	0.68	0.29	6.25	17.7	4.8	4.2	4.9
No. 3 " "	+23	14.7	53.0	1.08	0.43	8.24	20.4	12.1	9.9	10.2
No. 4 " "	+16	23.4	35.7	1.56	0.67	12.00	21.8	27.9	24.5	23.6
No. 5 " "	+12	16.4	21.7	1.83	0.80	15.50	9.3	22.9	20.5	21.4
Overflow**	-12	22.5	2.55	1.71	1.10	19.96	1.5	29.4	38.8	37.7
Total (calcd)		100.0	38.3	1.31	0.64	11.90	100.0	100.0	100.0	100.0

*for quartz particles
** Analysis calculated by difference.

TABLE 9

Metal Distribution in Flotation Tailing Cyclosizer Fractions

Size Fraction	Weight %	Analysis, %				Distribution, %			
		C	V	Ni	Si	C	V	Ni	Si
+250 mesh	0.5								
No. 1 cone u'flow	1.0	0.34	1.96	0.80	24.8	7.4	3.4	3.2	4.9
No. 2 " "	3.0								
No. 3 " "	8.7	0.15	2.40	0.99	21.2	7.1	9.2	8.6	9.1
No. 4 " "	25.2	0.10	2.32	1.02	20.0	13.6	25.7	25.6	24.8
No. 5 " "	25.6	0.07	2.29	1.02	20.5	9.7	25.8	26.0	25.9
Overflow	30.0	0.32	2.27	1.02	19.9	62.2	35.9	36.6	35.3
Total (calcd)	100.0	0.19	2.28	1.00	20.3	100.0	100.0	100.0	100.0
Total (assay)		0.21	2.02	1.10	20.1				

Mineralogical Examination

In order to determine the mode of occurrence of the constituents in the fly ash, sized fractions were submitted to the Mineral Sciences Division for mineralogical examination. This work is reported in detail in Internal Report MS-72-2 by D.R. Owen which is appended. The following is a summary of the results of mineralogical examination:

Microscopical and X-ray diffraction studies showed that the fly ash was composed of two types of material; amorphous carbon grains and amorphous, translucent colourless to brownish spheroids. Electron microprobe analysis indicated that the spheroids were essentially alumino-silicates containing varying amounts of Fe, Ca, Mg, Ni, V and S. It was not possible to relate variation in composition to colour differences in the spheroids. Most of the

carbon particles were concentrated in the coarsest size fractions.

Some of the fractions were also examined under a scanning electron microscope. Photomicrographs showed the porous nature of the carbon particles, Figure 2, and the almost perfect spherical shape of the silicates even down to a diameter of 1 micron (Figure 3).

Figure 2. Photomicrograph of Carbon Grains in Fly Ash, X200

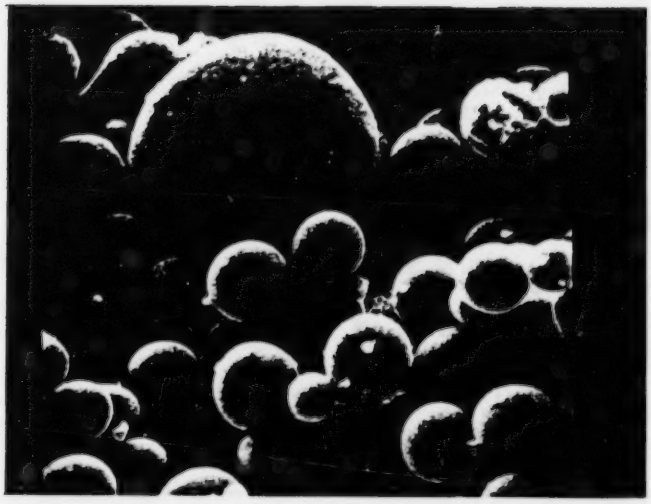


Figure 3. Photomicrograph of Silicate Spheroids in Fly Ash, X2000

CONCLUSIONS

Flotation is an effective method of removing the carbon from the fly ash. The carbon concentrate produced is equivalent in carbon content to the coke fed to the boilers and therefore could be recycled.

It was found necessary to use high amounts of kerosene and frothers in flotation probably because of the high adsorptive capacity of the porous carbon particles. The cost of kerosene would not be high as large quantities

of it are produced as an intermediate product in the refining process but cost of frothers would probably be the major cost for flotation.

Because most of the sulphur is associated with the carbon about 95% was recovered in the carbon concentrate. The resultant sulphur content in the tailing was much lower than could be obtained by roasting the fly ash. This gave the option of reducing the nickel to metal rather than recovering it in the form of a nickel matte.

Although mineralogical examination indicates that none of the vanadium and nickel is associated with the carbon, this is not confirmed by the per cent distribution of these metals in the pilot plant products given in Table 4. Note that about 7% of the silicate constituents (Si, Ca, Mg, Al) are distributed in the carbon concentrate as against 14% for vanadium and 11% for nickel. The difference could be due to a small content of these metals in the carbon particles.

Since practically all of the silica was present in the form of undifferentiated silicates and only an insignificant amount as free quartz, it was not possible to lower the silica content by flotation. At present a sample of coke is being examined with the point of view of determining the feasibility of removing some of the gangue (ash) prior to burning. This would result in a smaller volume of fly ash with a higher metal content and a consequent reduction in flux requirements for smelting.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the assistance of members of the staff of the Metallic Minerals Mill, Mineral Processing Division, who under the direction of Mr. A.J. Boisonnault set up the pilot plant equipment and assisted

in its operation.

They also wish to thank the following persons who made important contributions to the investigation:

Mr. J.C. Ingles, Head, Chemical Analysis Section of the Extraction Metallurgy Division under whose supervision most of the chemical analyses were carried out.

Mr. J.C. Hole of the Mineral Sciences Division who did the chemical analyses on products from initial batch flotation tests.

Mr. D.R. Owens of the Mineral Sciences Division who carried out the mineralogical examination of sized fractions of the fly ash and tailing.

Mr. G.A. Miller, Mineral Processing Division, who operated the scanning electron microscope and took the photomicrographs.

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APPENDIX

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MINERAL SCIENCES DIVISION

Internal Report MS-72-2

MINERALOGICAL EXAMINATION OF SIZED FRACTIONS OF A HEAD SAMPLE AND FLOTATION TAILING OBTAINED FROM FLY ASH ON BEHALF OF GREAT CANADIAN OIL SANDS LIMITED

by

D.R. Owens*

INTRODUCTION

Two sample of fly ash were received from Mr. A. Stemerowicz of the Mineral Processing Division on December 16, 1972. Mr. Stemerowicz stated that the fly ash, originally submitted to the Mines Branch by Great Canadian Oil Sands Limited, is a product recovered from the burning of petroleum coke. He requested that the sample be examined mineralogically to determine the mode of occurrence of the nickel, vanadium, and sulphur; the approximate quantity of discrete SiO_2 particles present; and the size range of the carbon particles and spheroids.

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SAMPLE

Two separate samples of fly ash were received from Mr. Stemerowicz. One consisted of a head sample which had been screened into plus 200, 200 to 400-, 400 to 500-, and 500-mesh size fractions. The other consisted of a flotation tailing, labelled "Test No. 3", which had been obtained from the fly ash by beneficiation methods. The flotation tailing had also been sized, by removing the plus 250-mesh fraction, and cyclosizing the remainder. These latter fractions were labelled as "cone underflow's Nos. 1, 2, 3, 4, 5, and final overflow". Analyses of both the head sample and the flotation tailing, before sizing, were provided by Mr. Stemerowicz. These are shown in Table 1.

TABLE 1

Chemical Analyses of the Head Sample and Flotation Tailing Before Sizing

Element	Head Sample Wt %	Flotation Tailing Wt %
Ni	0.66	1.10
V	1.40	2.02
Fe	4.11	6.90
Cu	0.0053	-
Ca	2.09	3.03
Mg	0.61	1.01
Si	11.60	20.10
Al	7.73	-
S	2.16	0.05
P	0.03	-
C	39.80	0.21

METHOD OF INVESTIGATION

One polished section was prepared from each sized fraction of both the head sample and the flotation tailing. These were examined microscopically to identify any metallic minerals that might be present. In addition, oil immersion mounts were prepared from each fraction, so that the gangue minerals could be studied and the proportions of carbon, quartz, and the spheroids could be estimated. X-ray diffractometer analyses were made on the three coarsest fractions of the head sample, to identify the major constituents and to determine their crystallinity. The distribution of the nickel, vanadium and sulphur in the fractions was determined by electron microprobe studies of the polished sections.

RESULTS OF INVESTIGATION

Characteristics of the Head Sample

Microscopical and X-ray diffraction studies show that the sized fractions of the head sample consist of amorphous carbon; amorphous, translucent, colourless to brownish spheroids; and up to 0.5 per cent quartz and iron oxides. Estimates of the quantities of amorphous carbon and spheroids in each fraction are shown in Table 2, and their distributions, observed in an oil immersion mount of the 400- to 500-mesh fraction, is shown in Figure 1.

TABLE 2

Estimates of Quantities of Amorphous Carbon and Spheroids in the Sized Fractions

Mesh Size	Per Cent Carbon	Per Cent Spheroids
+200	98	1 to 2
-200+400	95	5
-400+500	60 to 65	35 to 40
-500	10 to 15	85 to 90

Electron microprobe studies show that the spheroids are essentially alumino-silicates containing varying amounts of Fe, Ca, Mg, Ni, V and S, but it was not possible to determine their compositions quantitatively. These studies also show that the amorphous carbon grains contain significant amounts of sulphur.

Characteristics of the Flotation Tailing

The sized fractions of this sample differ from those of the head sample in that the presence of black amorphous carbon grains, with one exception, is negligible. The exception is the plus 250-mesh fraction which was screened from the flotation tailing before it was cyclosized. This fraction contains approximately 60 per cent carbon, 20 per cent quartz, 20 per cent spheroids, a few granular alumino-silicates, and several grains of an iron oxide which contains a few small inclusions of what is believed to be chalcopyrite (?).

The remaining fractions are composed almost entirely of spheroids (Figure 2), and contain trace amounts of quartz and iron oxides. A few particles of carbon occur in the coarsest fractions, but the amounts are less than 0.5 per cent. As mentioned above, the spheroids vary in

colour from almost colourless to dark brown. Whether this colour difference is due to variations in composition of the spheroids could not be determined.

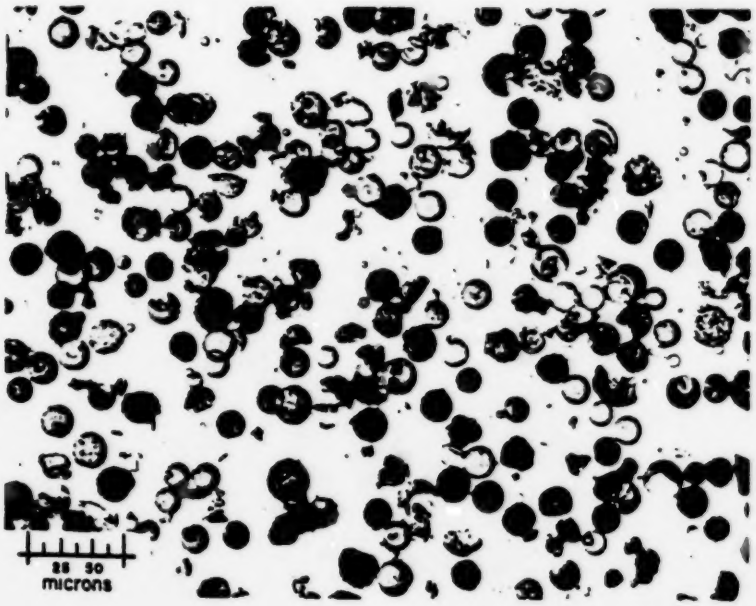

Figure 1. Photomicrograph of an oil immersion mount of the 400 to 500-mesh fraction of the head sample showing the proportions of amorphous carbon grains (black) to spheroids (light grey).

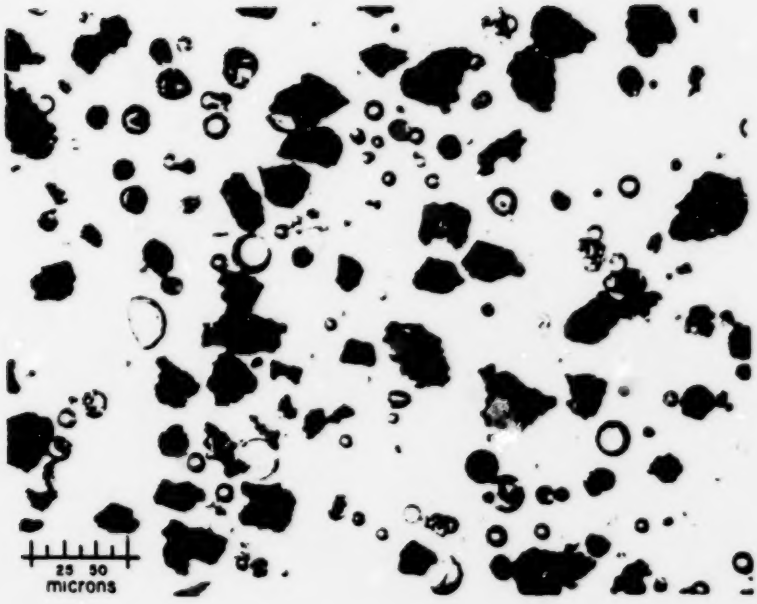

Figure 2. Photomicrograph of an oil immersion mount of the No. 3 Cone Underflow showing the concentration of spheroids of various shades.

Distribution of the Nickel, Vanadium, Sulphur and Silica

Nickel and vanadium were found only as constituents of the amorphous spheroids, and of the few granular alumino-silicates grains. Electron microprobe studies show that the spheroids contain approximately 0.9 to 1.2 per cent nickel, and 1.7 to 5.4 per cent vanadium.

The majority of the sulphur occurs as a constituent of the amorphous carbon, but trace amounts (0.0 to 0.15 per cent) are present in the spheroids and as chalcopyrite (?) inclusions in the iron oxides. The sulphur content of the carbon particles is somewhat variable, but it is generally close to eight per cent.

It is estimated that most of the silica is present as a constituent of the amorphous spheroids, and that the amount present as quartz is negligible. The quartz content of all the fractions, except one, is approximately 0.5 per cent or less. The exception, is the plus 250-mesh fraction screened from the flotation tailing before it was cyclosized. This fraction contains an estimated 20 per cent quartz.

ACKNOWLEDGEMENTS

The author wishes to express his appreciation to Mr. E.J. Murray for the X-ray diffractometer analyses; to Mr. R.G. Pinard for the photomicrographs; and to Mr. Y. Bourgoin for the preparation of the polished sections.

CONCLUSIONS

The mineralogical examination of the sized fractions of the head sample and flotation tailing leads to the following conclusions.

1. The nickel and vanadium are present almost exclusively as constituents of the amorphous spheroids, with negligible amounts present in a few grains of granular alumino-silicates.
2. Sulphur is concentrated in the amorphous carbon, but minute amounts are present in the spheroids, and as chalcopyrite (?).
3. Most of the silica occurs as a constituent of the amorphous spheroids. Minute amounts are present as granular alumino-silicate grains, and as quartz.
4. The majority of the carbon particles are concentrated in the two coarsest fractions of the head sample.

DRO/pg

POOR

QUALITY

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